

NASA Technical Memorandum

104096

IN-27
61456
P 28

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(NASA-TM-104096) AN ANALYSIS OF
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BLANKET MATERIAL (NASA) 28 p CSCL 11C

N92-15189

Unclass

G3/27 0061456

December 1991



National Aeronautics and
Space Administration

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ABSTRACT

The characterization of selected silvered fluorinated ethylene propylene (FEP) teflon thermal blanket material which received 5 years and 9 months of exposure to the LEO environment on the Long Duration Exposure Facility is reported. X-ray photoelectron spectroscopy, infrared, and thermal analyses did not detect a significant change at the molecular level as the result of this exposure. However, various microscopic analyses revealed a roughening of the coating surface due to atomic oxygen erosion which resulted in some materials changing from specular reflectors of visible radiation to diffuse reflectors. The potential effect of silicon-containing molecular contamination on these materials is addressed.

1. INTRODUCTION

The National Aeronautics and Space Administration's Long Duration Exposure Facility (LDEF) provided a unique environmental exposure of a wide variety of materials and experiments (1,2). The spacecraft traveled approximately 33,000 orbits and three-quarters of a billion miles during its 5-year and 9-month journey. The effects of atomic oxygen, ultraviolet and particulate radiation, meteoroid and debris, vacuum, contamination and thermal cycling on the LDEF and its contents is providing a data base unparalleled in the

history of space environmental research. This paper reports on the analysis of selected silvered FEP teflon thermal blanket material which flew onboard the spacecraft.

The LDEF structure and orbital orientation is depicted in Figure 1. Preliminary environmental exposure conditions are summarized in Table I. The spacecraft was 30 feet long, 14 feet in diameter, and had 12 rows with 6 experiment trays per row (1). Additional experiment trays were mounted on the earth and space ends. The orientation was such that Row 9 nominally faced the RAM direction,* Row 3 the WAKE direction, one end always pointed toward space, and the other end always pointed toward Earth. Thus, LDEF provided a stable exposure platform. The environment a specimen experienced depended on its location on the vehicle. The silvered FEP teflon specimens examined in this study came from thermal blankets which provided thermal protection for experiments located at Tray F on Row 2 (Tray F2) and Trays C5, C8, and A10. Additional adhesively bonded silvered FEP teflon specimens were obtained from LDEF experiments on Trays B9 and F9. Thus, these specimens received essentially RAM to WAKE exposure.

The blanket material, also known as flexible second-surface mirror (SSM) thermal control coating, is used in a variety of space applications where thermal protection is a consideration (3). Figure 2 gives a schematic of the SSM and describes its composition and function. The coating consists of a nominally 5 mil perfluorinated ethylene propylene copolymer (FEP) Type A Teflon film covered on the back side with approximately 1600Å of vapor deposited silver and then 400Å of vapor deposited inconel. When used as a thermal blanket, a 3-5 mil layer of black urethane-base paint is applied to the metallized side. In other applications, the coating may be adhesively applied to another surface. Solar radiation passes through the transparent film and is reflected away by the specular silver backing. This yields a low absorption of solar radiation, or α_s . The high infrared thermal emittance, ϵ , of the outer

*Recent LDEF supporting data analyses have determined that the actual RAM direction was 8° of yaw from the perpendicular to Row 9, in the direction of Row 10.

polymer surface is efficient in radiating thermal energy. Thus, this coating produces a low α_s/ϵ ratio, desirable in certain spacecraft applications.

The visible appearance of the silvered FEP materials which flew on LDEF changed depending on the atomic oxygen fluence they experienced. Several analytical techniques including X-ray photoelectron spectroscopy, infrared spectroscopy, thermal analysis, and selected microscopic analyses were used to characterize flight specimens. The objective of the present research was to assess the response of selected samples to the extended low Earth orbital environment. This report is intended to add to the body of knowledge on space environmental effects on materials being derived from the LDEF mission.

2. EXPERIMENTAL

Materials came from various thermal blanket specimens made available to the Materials Special Investigation Group during LDEF deintegration activities at the Kennedy Space Center during the January-May 1990 time period. Samples were cut to contain a particular item of interest, such as opaque or specular areas, visible contamination, or a micrometeoroid impact. They were placed in containers, identified by LDEF tray and row, and taken to the Langley Research Center during Spring 1990. Specimens were then stored in a low-humidity environment until analyzed.

X-ray Photoelectron Spectroscopy (XPS) measurements were conducted under NASA Grant NAG-1-1186 at the Virginia Tech Surface Analysis Laboratory, Department of Chemistry, VPI&SU, Blacksburg, VA. Measurements were made on a Perkin-Elmer PHI 5300 spectrometer with Mg $K\alpha$ source (1253.6 eV) operating at 15 kV with an emission current of 20 mA. Typical operating pressures were $<10^{-7}$ torr. Analyses were made at take-off angles of 45° or 90°.

Scanning electron microscopy (SEM) and energy dispersive X-ray (EDS) analyses were conducted both at Virginia Tech and at Langley. An ISI SX-40 SEM (ISI, Milpitas, CA) equipped with a Tracor Northern Z-MAX 30 EDS analyzer (Tracor, Madison, WI) was used

at Virginia Tech. A Cambridge StereoScan 150 SEM (Cambridge Instruments, Deerfield, IL) equipped with an EDAX S150 detecting unit (EDAX International Inc., Prairie View, IL) was employed at the Langley Research Center.

Scanning tunneling microscopy (STM) was performed on a NanoScope II instrument (Digital Instrument, Inc., Santa Barbara, CA). UV-VIS reflectance spectra were recorded on a Beckman DK-1A equipped with a Gier-Dunkle Integrating Sphere (4). Infrared spectra were recorded on a Nicolet 60SX Fourier Transform Infrared Spectrometer System using a diffuse reflectance technique (5). Thermal analyses were performed on a DuPont Model 1090 Thermal Analyzer/Model 910 Differential Scanning Calorimeter (DSC). The visual appearance of selected specimens was documented using various photographic techniques.

3. DISCUSSION

Visual inspection of the fully integrated LDEF in the Spacecraft Assembly and Encapsulation Facility (SAEF-II) at the Kennedy Space Center confirmed observations made during orbital retrieval that the appearance of the silvered FEP teflon thermal blankets was not the same at all locations. Blankets located near the Row 3 trailing edge, for example on Trays A2, E2, A4, and F4, exhibited a highly specular appearance. Blankets located near the Row 9 leading edge, for example on Trays C8, A10, and E10, exhibited a diffuse or "frosted" appearance. Figure 3 illustrates this for F4 and E10. In addition, the change from specular to diffuse was essentially graduated as the location changed from trailing edge to leading edge. Since this observation correlated with the anticipated atomic oxygen fluence at those locations, the two phenomenon were assumed to be related.

3.1 Chemical Characterization

The increase in the diffuse component of the silvered FEP teflon upon exposure to the LDEF environment is depicted in Figure 4. This figure shows UV-VIS-NIR reflectance spectra for an adhesively bonded control specimen and an identical specimen which received 5.8 years of LDEF exposure on a Row 9 experiment (6). Figure 5 shows a photograph

of the flight-exposed specimen. This specimen exhibited a significantly increased diffuse component, especially in the visible region. This phenomenon had been previously noted on an STS-8 experiment (7). Surprisingly, the total reflectance (diffuse plus specular) of the two specimens is virtually the same. The latter phenomenon resulted in no measurable difference in the absorptivity to solar radiation (α_s) for these two specimens.

The origin of the observed change in appearance focused on several speculations. One explanation that was actively pursued in this research was the possibility that highly reactive atomic oxygen had entered into the surface chemistry of the FEP layer to change its molecular structure and/or morphology. Thus, XPS was used to examine various exposed specular and diffuse specimens, as well as several standard and control materials. Table II summarizes results for selected specimens.

A comparison of data for specular standard and control samples with data for opaque A10 and C8 specimens, suggests no significant change in surface chemistry as the result of exposure. No appreciable amount of oxygen was incorporated into these materials nor had the surface concentration of carbon and fluorine changed. Figure 6 gives narrow scan XPS spectra for C8 showing peaks for carbon, oxygen, and fluorine. These spectra are superimposable on spectra obtained for control and standard samples. Thus, XPS results showed no real difference in surface chemistry as the result of environmental exposure and indicated that opaqueness did not develop as the result of changes in the chemical properties of the FEP teflon layer.

As will be discussed in a later section, the surface of most XPS-examined flight specimens that remained specular was found to contain a molecular contamination. Thus, data for these specimens are not included in Table I since the measured atomic concentrations for C, O, and F do not reflect the surface chemistry of the FEP teflon layer.

Additional chemical characterization did not reveal any difference between exposed opaque and specular samples and material that was not flown. Figure 7 gives FTIR spectra of C8, C5, and F2 control materials. Although the diffuse reflectance spectra are not highly

resolved, they appear to be essentially identical. Various subtractive techniques revealed only differences that could be attributed to contamination. Thermal analysis of selected blanket materials also failed to detect significant differences. The FEP film layer was delaminated from the urethane backing and analyzed. The vapor deposited silver remained on the urethane layer. Figure 8 shows the DSC thermogram from -120°C to 340°C for the C5 sample. Inflections in the trace around -10°C are likely associated with the glass transition of the FEP teflon. The melt endotherm around 250°C is also apparent. All analyzed specimens showed essentially the same DSC thermogram. No significant differences were noted in T_g , T_m , or the heat of fusion. In addition, no interpretable differences were noted when the urethane layer was analyzed by this technique.

3.2 Microscopy

SEM showed physical differences at the microscopic level between opaque and specular thermal blanket materials. Figure 9 compares photomicrographs of C5 (specular) and C8 (diffuse) flight specimens at three different magnifications. Considerable surface erosion or texturing is noted with the C8 material, which received approximately nine orders of magnitude greater atomic oxygen fluence than did C5. The SEM of the A10 thermal blanket, given in Figure 10, also shows severe surface erosion. This type of surface behavior had been previously documented when various polymeric materials received exposure on earlier shuttle flights (8). However, the extent of surface erosion of LDEF FEP teflon materials probably was not anticipated.

Preliminary estimates are that the FEP film lost in excess of 0.001 inch of its original 0.005 inch thickness in the most extreme (Row 8-10) cases. A uniform erosion of the film surface due to atomic oxygen impingement might be intuitively expected. However, SEM analysis of C8 in Figure 9 and A10 in Figure 10 shows that this erosion was not uniform. The peaks and valleys along the surface have been described as a "carpet" morphology (8). A superficial explanation for this texturing is that the atomic oxygen-eroded portion of the

film surface may have become sensitized to further AO erosion, producing the "carpeted" surface.

Scanning tunneling microscopy (STM) analysis proved to be effective in profiling the surface of control and exposed materials. Figures 11-13 show a series of STM line plots for control, C5, F2, and C8 specimens. Further discussion of these figures is merited.

The scanned area for the control, C5, and F2 specimens was 12,000 nm on a side. A z-direction scale is included in the 3-dimensional drawings. The C8 specimen was so rough that a smaller area was selected for analysis.

STM analysis of the specular control specimen in Figure 11 revealed a smooth surface. The specular C5 sample, which experienced limited AO exposure, was also relatively smooth. The marked texturing in the F2 specimen is depicted in Figure 12. The particular F2 sample selected for STM characterization was taken from a slightly diffuse-appearing portion of the thermal blanket; the majority of the material was specular. The analysis of the C8 specimen shown in Figure 13 revealed a very rough surface. The area scanned had to be decreased and the z-direction increased to keep the STM probe from contacting the surface during analysis. Even with this precaution, the analyst had to "pick" the portion of the sample that was actually analyzed. Large surface projections were observed. FEP teflon specimens taken from Row 9 and Row 10 experiments could not be analyzed by this technique due to the probe "crashing" as it scanned the roughened surface.

Exposed diffuse specimens were observed to take on a specular appearance when the surface was disturbed by scraping with a blunt object. Figure 13 also shows the STM of a C8 specimen brought back to specularity by rubbing several times with the edge of a spatula. Surface projections appear to have been smoothed or sheared off by this action. XPS data showed that the chemistry of the silvered FEP teflon returned to a specular state by rubbing had not changed. Table II contains this data for a C8 specimen after the surface was wiped with a paper towel containing ethanol. The abrasive action brought the sample back to

a specular state. Carbon and fluorine concentrations for the then-specular specimen were essentially the same as that for other samples in Table II.

The possibility that a small amount of oxygen may actually have been incorporated into the FEP teflon due to exposure cannot be ignored. Table III gives XPS data for adhesively bonded silvered teflon specimens which received 10 months of exposure on a B9 experiment, 5.8 years of exposure on that same experiment, 5.8 years of exposure on an F9 experiment, and a control specimen. Data on the A10 thermal blanket specimen are also included in the table. These samples experienced the highest AO flux due to their positions on Row 9 and Row 10 of the LDEF. Up to about 1.5% oxygen was detected at the surface of these films; no elements indicative of contamination were noted which might explain this presence of oxygen. How these oxygen atoms might be attached to the FEP molecular structure was not determined in this study.

Based on the characterization of LDEF-exposed silvered FEP teflon specimens examined in this study, the following preliminary explanation of their appearance is offered. Chemical analysis of control, exposed specular, and exposed diffuse specimens revealed no significant differences at the molecular level. This conclusion is based on the results of XPS, infrared, and thermal analyses. Thus, the phenomenon likely does not arise from a change in polymer chemistry.

Microscopy revealed considerable surface texturing or carpeting of AO exposed opaque specimens. STM analysis showed that the order of magnitude of this texturing was the same as that of the wavelength of visible light. An examination of Figure 12 will support this conclusion. The opaqueness of exposed materials is likely due to the interaction of visible light with the roughened surface through classical reflection-refraction processes. When the surface is smooth, either because it saw no significant AO fluence or was rubbed as in Figure 13, there is no interaction and the material is specular. Thus, the phenomenon arises from a change in physical properties rather than a change in chemical properties.

3.3 Contamination

A thin, transparent, amber-colored film covered selected areas of some of the thermal blanket specimens, especially around the outer perimeter where edges were tucked inward when the blanket was placed over the experiment tray. Figure 14 shows an SEM of a visibly-stained portion of the A10 thermal blanket. The amorphous-appearing film is fractured when viewed under magnification. Table IV gives the results of XPS analyses of this material. Two analyses were made, one directly on the stain and one at a distance from the visible stain where the blanket appeared uncontaminated. The two analyses were essentially the same, suggesting that the contamination covered a larger area than was visible to the naked eye. About 20% silicon was found on these specimens.

Approximately 1.5% silicon was also found on the surface of the F2 blanket specimen. Possibly because of this contamination, the measured atomic concentrations of C, O, and F obtained by XPS did not match control values. Table IV gives this data for F2 along with results for C5. Although no silicon was found on the latter specimen, C, O, and F values also did not conform with control values. Figure 15 shows the narrow scan XPS analysis of C5. At least three different chemical states for carbon were detected. Subtractive DR-FTIR techniques confirmed the presence of a carbonyl-containing species on this particular specimen. Emerging research is suggesting that the FEP surface is crosslinked by deep UV exposure (9). The possibility of UV crosslinking of fluorinated polymers is currently being investigated under NASA Grant NAGW-2495 at the University of Queensland, Queensland, Australia. This phenomenon likely would not be observed with opaque blanket specimens because atomic oxygen would have eroded the UV-crosslinked surface away.

The amber-colored silicon-containing contamination may have resulted from outgassing of the adhesive which secured velcro hook and loop tape onto the thermal blankets which, in turn, secured the thermal blanket on the experiment tray. These pads, some as large as 1 inch by 4 inches, were bonded with DC6-1104 RTV silicon adhesive. A visual inspection of two velcro strips on a section of blanket showed that the adhesive had been liberally applied.

Approximately 50 pads were attached to the blanket material. A matching set of pads were bonded to the tray itself. Thus, a significant amount of this silicon adhesive was used in this particular application, since at least 16 thermal blankets were held in place using this technique (10).

The silicon from this source, perhaps in the form of an organic silicone, probably contributed to the general molecular contamination observed at various locations on LDEF experiments and structure. The potential significance of this particular contaminant is the possibility of conversion to an inorganic silicate due to reaction with atomic oxygen (11). Silica/silicates have been shown to be effective barriers to AO erosion (8,12,13). Thus, surfaces which were covered with this contaminant may have responded differently to the LDEF environment than surfaces which were not contaminated.

CONCLUDING REMARKS

The LDEF is providing a wealth of information on the effects of extended exposure of spacecraft materials to the space environment. The present study examined how silvered FEP teflon thermal blanket material flown on LDEF changed after almost 6 years of exposure in low Earth orbit. Although a limited number of specimens were examined, the pattern which emerged suggested that extended LEO exposure did not significantly alter the chemistry of the FEP teflon layer. However, it did result in a change in the physical properties of that surface. Depending on the atomic oxygen fluence, the silvered FEP teflon second-surface mirror coating changed from a specular reflector of radiation to a diffuse reflector due to a roughening of the surface. A silicon-containing molecular contamination was also observed on selected specimens. The characterization of a larger sample of this thermal blanket material, possibly including mechanical property information, is anxiously awaited. The ultimate benefit will be a more fundamental understanding of space environmental effects and an increased confidence in future spacecraft materials and design.

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TABLE I PRELIMINARY ENVIRONMENTAL EXPOSURE CONDITIONS

- Atomic oxygen
0 to 10^{22} atoms/cm²
(wake to ram)
- UV radiation
100-400 nm; 16,000 hrs
- Particulate radiation
e⁻ and p⁺: 2.5×10^5 rad
surface fluence
Cosmic: <10 rads
- Micrometeoroid and debris
6000 particles from
0.1 mm to 2 mm
- Vacuum
 10^{-6} - 10^{-7} torr
- Thermal cycles
~34,000 cycles: -20 to
190°F, $\pm 20^\circ$
- Altitude
255-180 nautical miles
- Orbital Inclination
28.5°

TABLE II XPS RESULTS FOR SELECTED SILVERED FEP TEFLON THERMAL BLANKET MATERIALS

PHOTO PEAK	STANDARD ^a	CONTROL ^b	A10 OPAQUE	C8 OPAQUE	C8 SPECULAR ^c
C 1s B.E. (eV) ^d	290.9	290.9	290.9	290.9	290.9
AC (%) ^e	31.6	29.6	28.5	30.4	32.1
F 1s B.E.	689.0	688.9	688.5	688.8	688.7
AC	65.6	66.4	69.8	66.2	64.5
O 1s B.E.	---	---	530.9	---	NSP
AC	NSP ^f	<1.0	1.3	<0.5	---
Na 1s B.E.	---	---	---	---	---
AC	NSP	NSP	NSP	NSP	NSP
Be 1s B.E.	---	---	---	---	---
AC	NSP	NSP	NSP	NSP	NSP
Al 2p B.E.	---	---	---	---	---
AC	NSP	NSP	NSP	NSP	NSP
Ag 3d ⁵ B.E.	---	---	---	---	---
AC	NSP	NSP	NSP	NSP	NSP
Si 2p B.E.	---	---	---	---	---
AC	NSP	NSP	NSP	NSP	NSP

^a Commercially obtained

^b Flight control blanket.

^c After scraping surface.

^d Binding energy, electron volts.

^e Atomic concentration, percent.

^f No significant peak.

**TABLE III XPS RESULTS FOR ROW 9 AND ROW 10
SILVERED FEP TEFLON MATERIALS**

PHOTO PEAK	STANDARD/ NONE ^a	SPECIMEN/LDEF EXPOSURE			
		B9/10 mo. ^b	B9/5.8 yr. ^c	F9/5.8 yr. ^c	A10/5.8 yr. ^d
C 1s B.E. (eV) ^e	290.9	290.9	290.9	290.9	290.9
A.C. (%) ^f	31.3	30.8	31.4	38.1	28.5
F 1s B.E.	688.6	688.4	688.6	688.6	688.5
A.C.	68.7	68.7	67.5	59.2	69.8
O 1s B.E.	--	532.5	532.2	531.6	530.9
A.C.	NSP ^g	0.5	1.1	1.6	1.3
Si 2p B.E.	--	--	--	--	--
A.C.	NSP	NSP	NSP	NSP	NSP

^a Adhesively bonded, Langley archived.

^b Adhesively bonded, environmental control canister exposed.

^c Adhesively bonded, full exposure.

^d Thermal blanket specimen.

^e Binding energy, electron volts.

^f Atomic concentration, percent.

^g No significant peak.

**TABLE IV XPS RESULTS OF CONTAMINATED
SILVERED FEP TEFLON THERMAL
BLANKET MATERIALS**

PHOTO PEAK	A10		F2	C5
	STAINED	UNSTAINED		
C 1s B.E. (eV) ^a	284.6	284.6	290.9/289.0/284.2	290.9/289.0/284.5
A.C. (%) ^b	31.7	30.8	39.8	37.5
F 1s B.E.	689.3	689.6	686.8	686.9
A.C.	1.2	2.3	47.8	56.5
O 1s B.E.	533.0	532.8	531.0	531.0
A.C.	47.9	46.5	7.4	2.8
Si 2p B.E.	103.4	103.2	102.7	--
A.C.	19.2	20.5	1.5	NSP ^c

^a Binding energy, electron volts.

^b Atomic concentration, percent.

^c No significant peak.

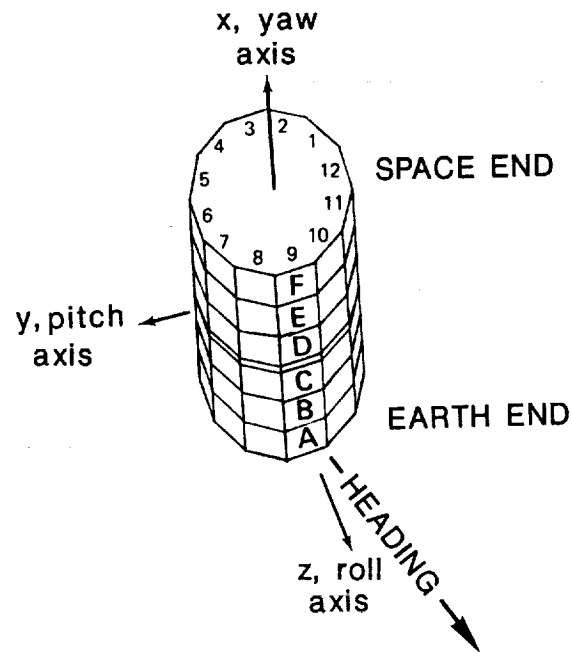


Figure 1. LDEF sketch and orbital configuration.

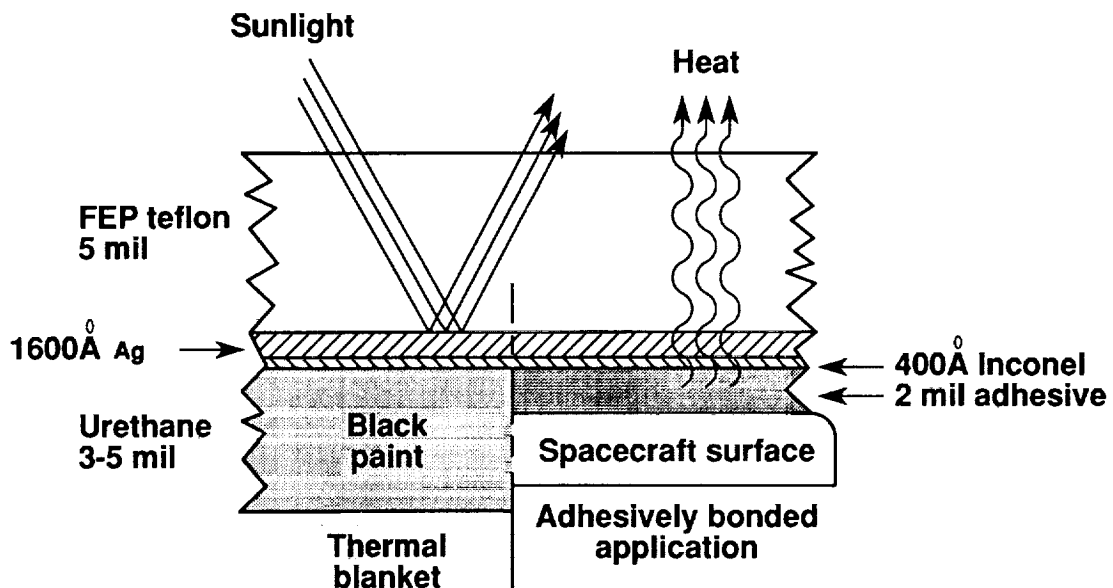
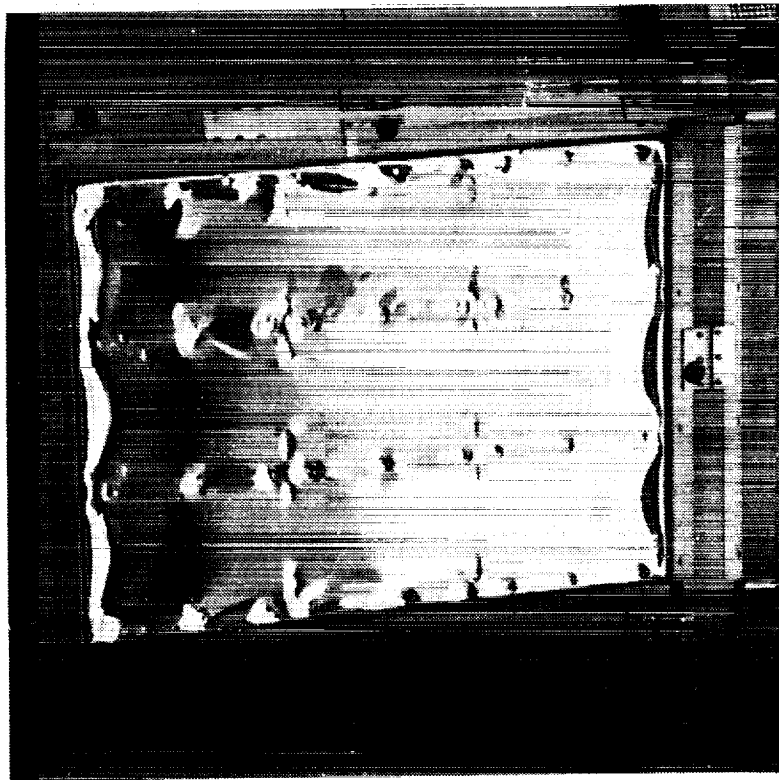
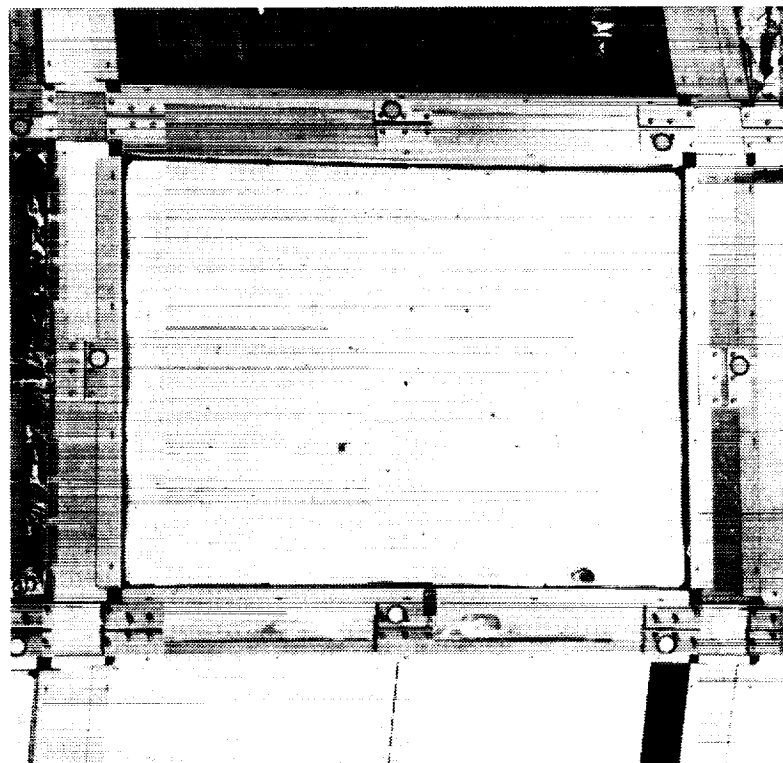


Figure 2. Flexible second-surface mirror thermal control coating in thermal blanket and adhesively bonded applications.



(a) F4 thermal blanket.



(b) E10 thermal blanket.

Figure 3. On orbit retrieval photographs of (a) F4 and (b) E10 silvered FEP teflon thermal blankets.

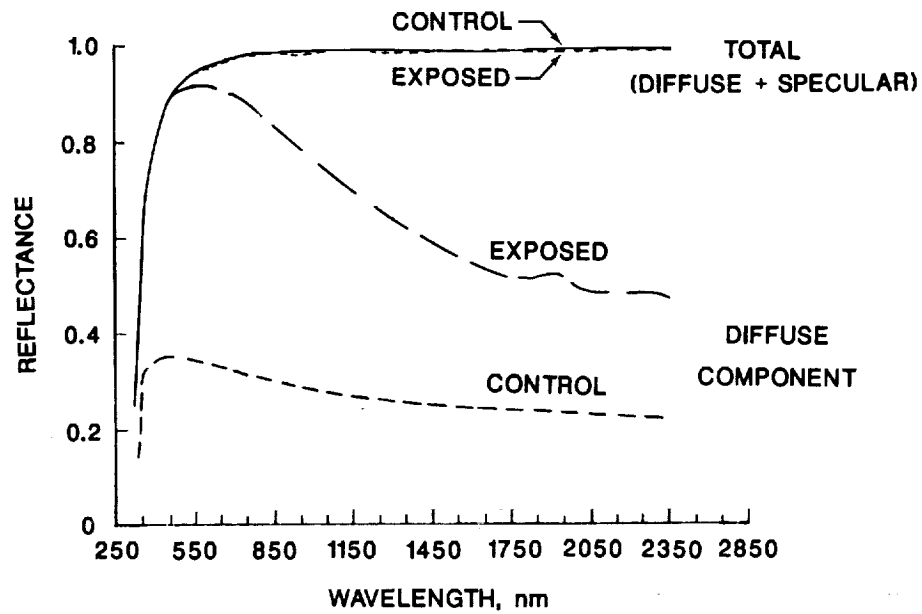


Figure 4. Reflectance spectra of control and LDEF exposed adhesively bonded silvered FEP teflon specimens.

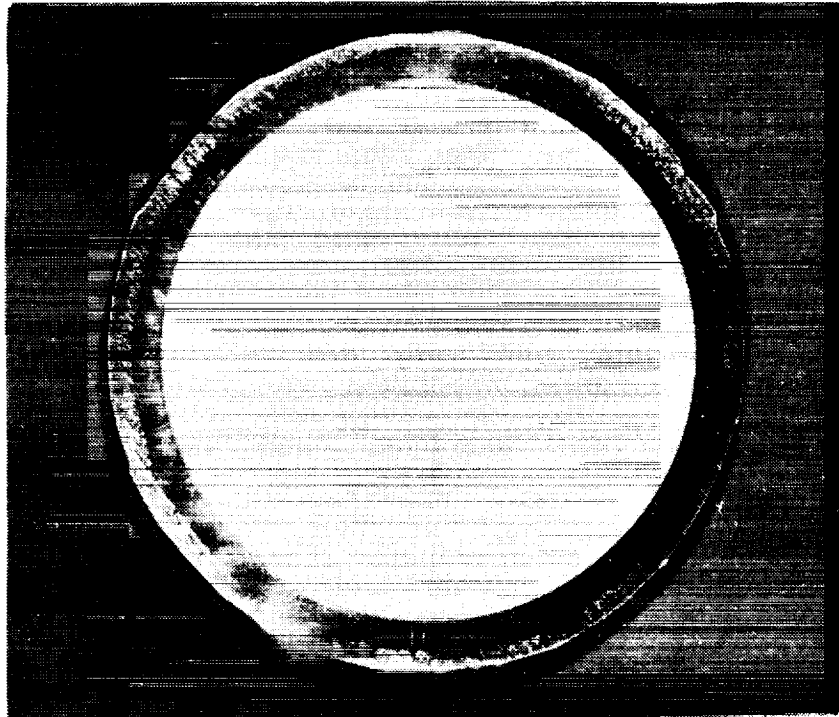


Figure 5. Photograph of adhesively bonded silvered FEP teflon specimen after 5.8 years of LDEF exposure.

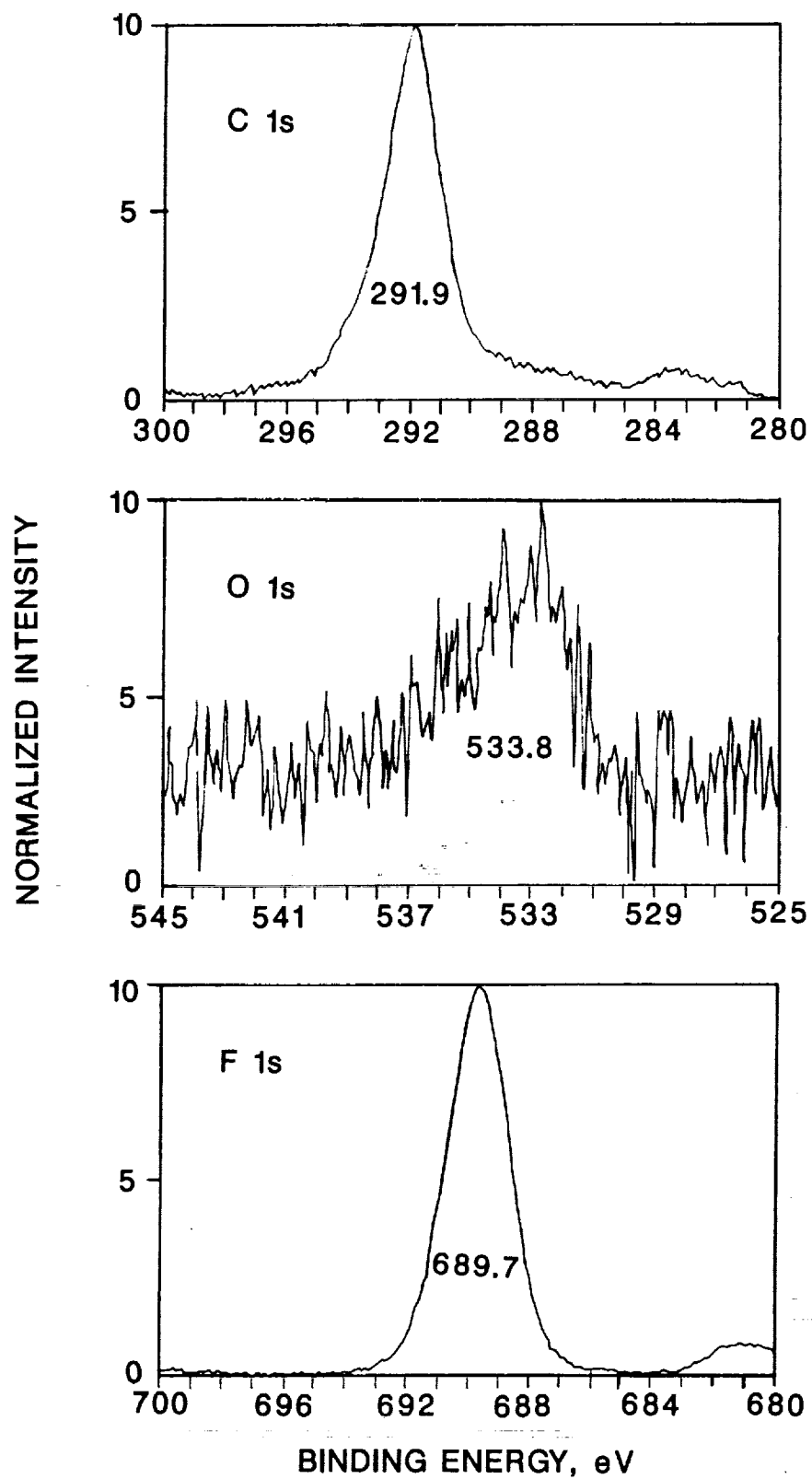


Figure 6. Narrow scan XPS spectra for C8 thermal blanket specimen.

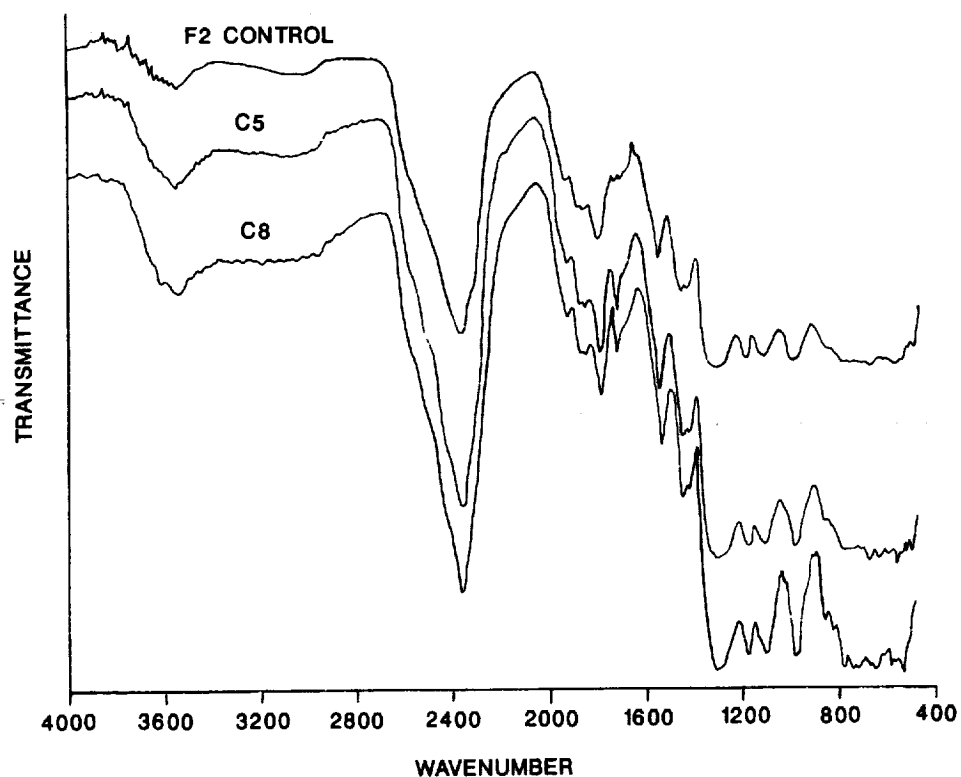


Figure 7. Diffuse reflectance-FTIR spectra of F2 control, C5 and C8 thermal blanket specimens.

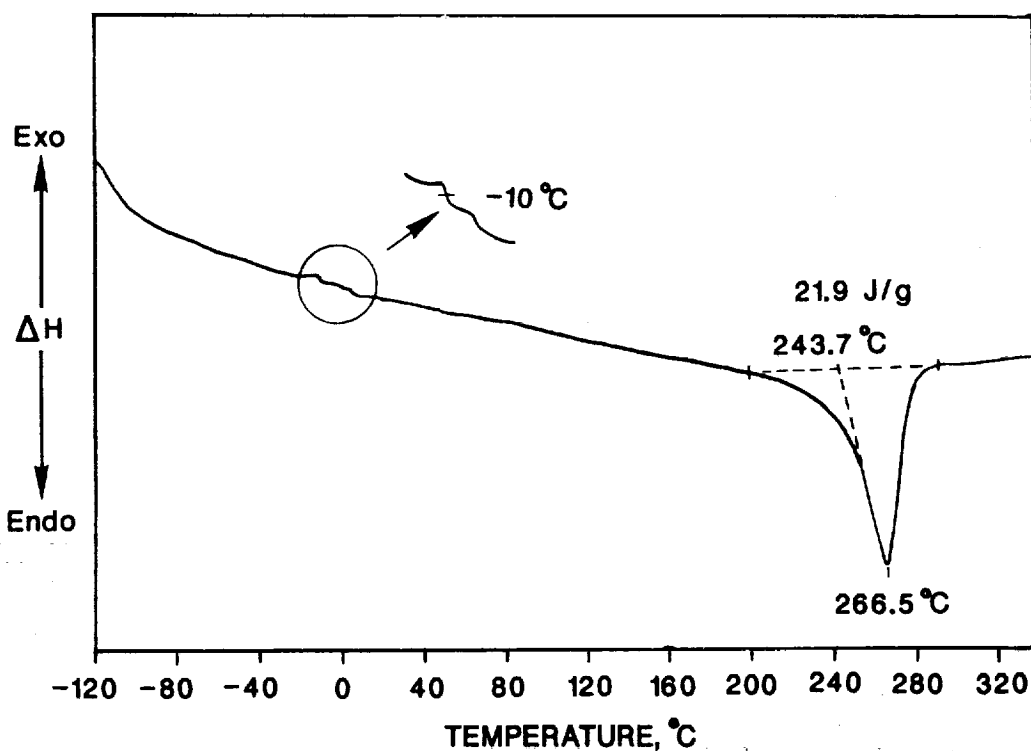


Figure 8. DSC thermogram of C5 thermal blanket specimen.

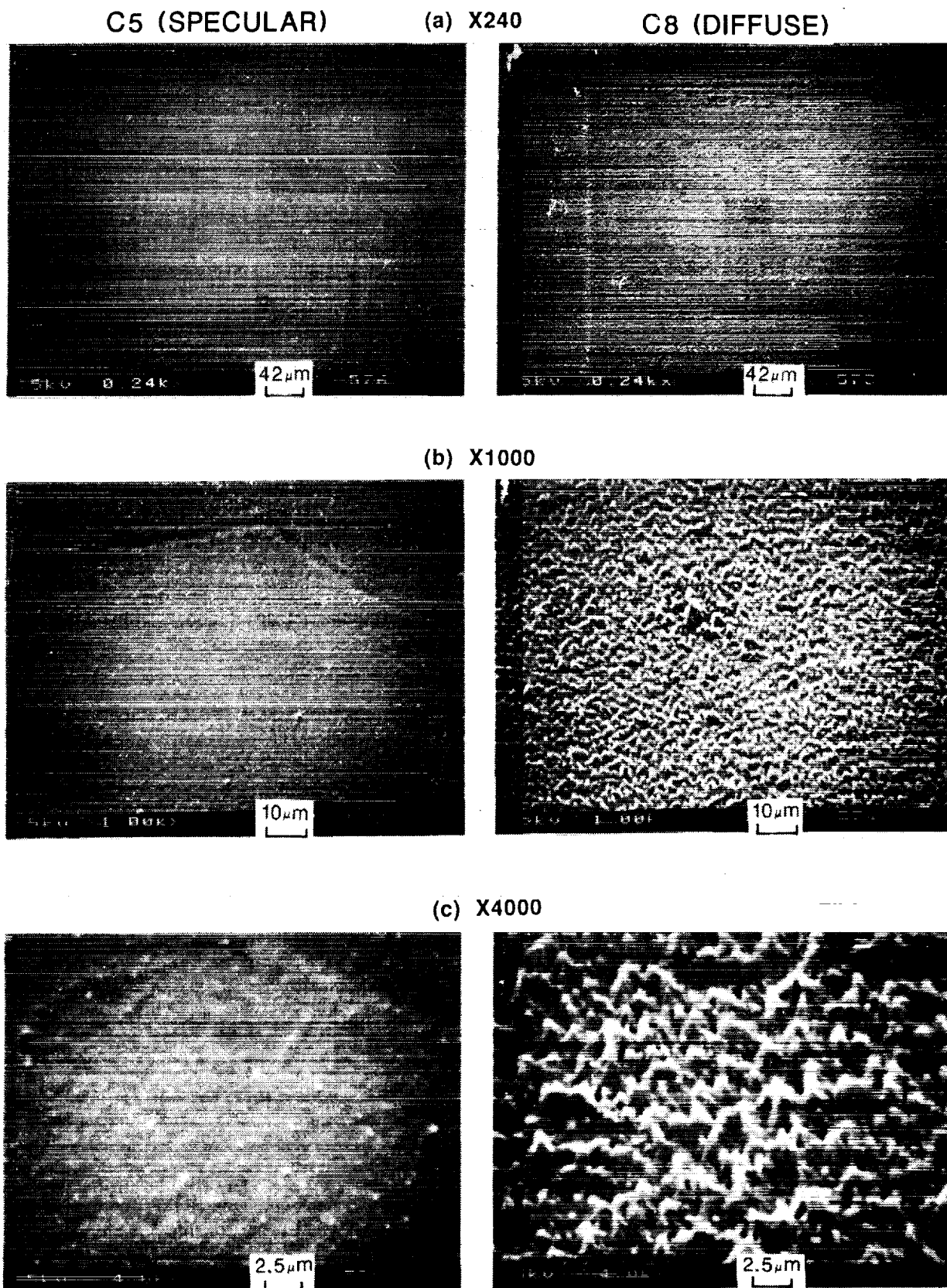
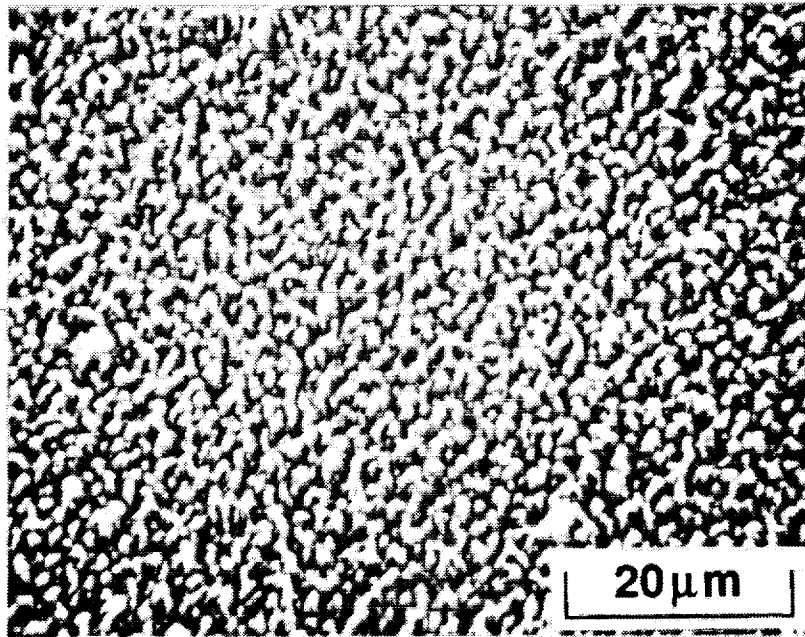


Figure 9. SEM of C5 and C8 silvered FEP teflon thermal blanket specimens at three magnifications.

X 1500



X 3000

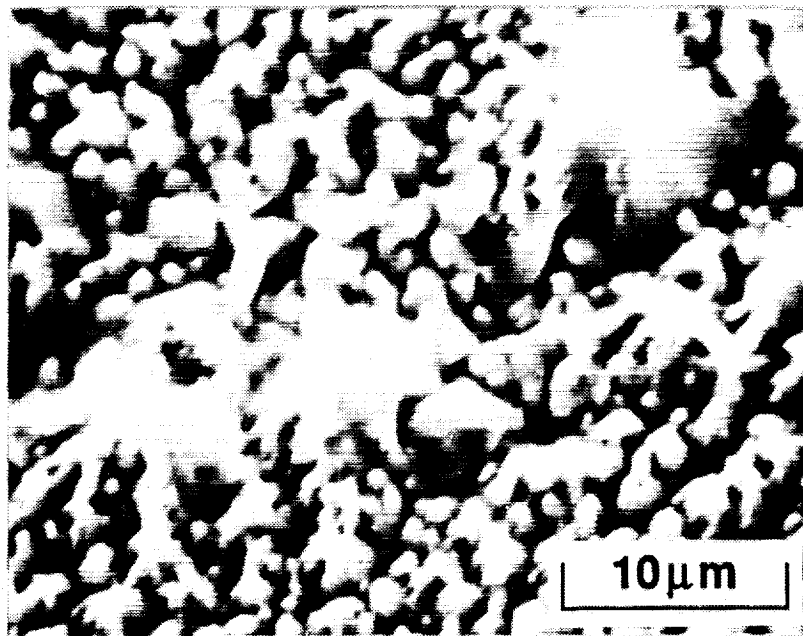
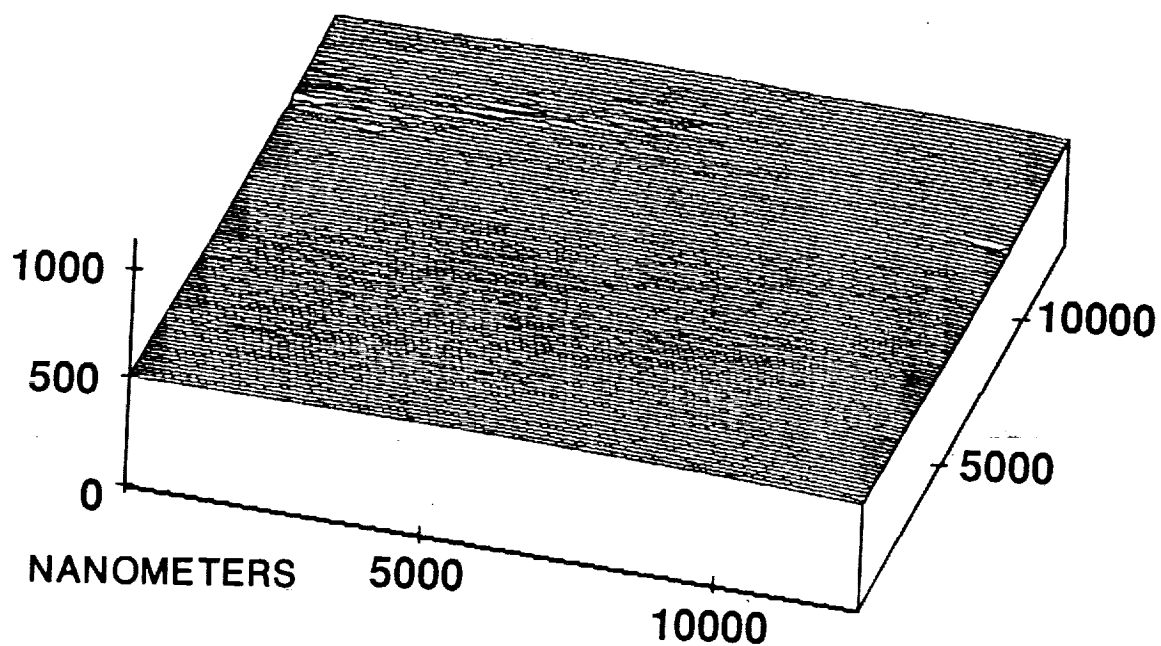
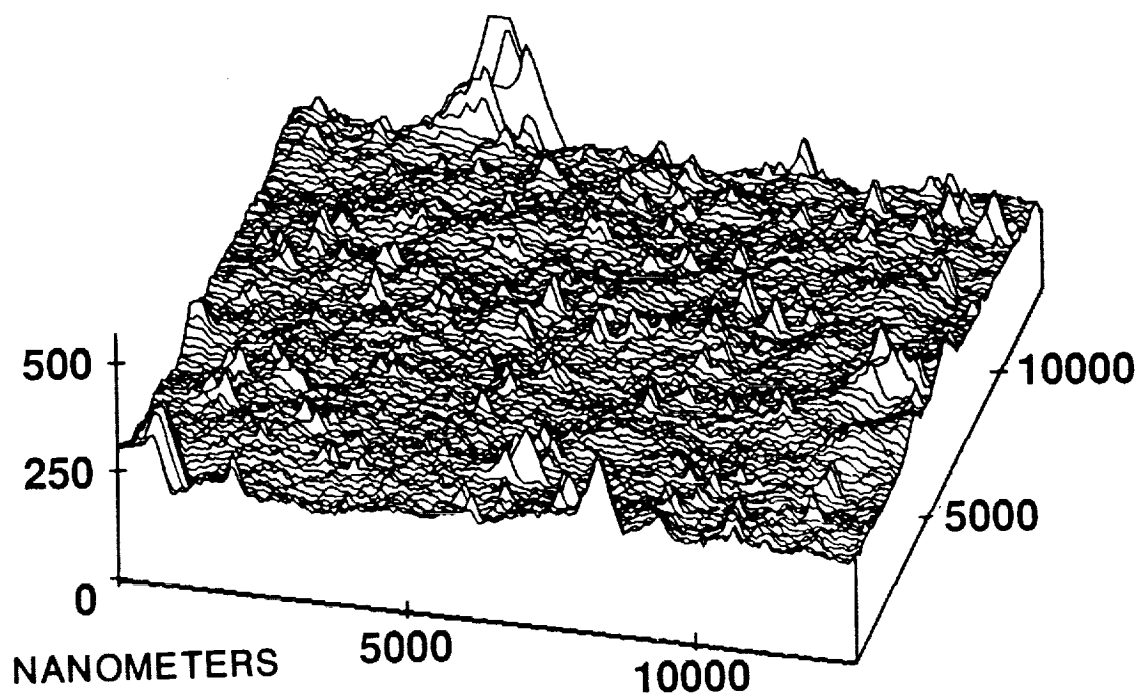


Figure 10. SEM of A10 silvered FEP teflon thermal blanket specimen at two magnifications.



(a) control.



(b) LDEF exposed C5.

Figure 11. STM line plot analysis of (a) control and (b) C5 silvered FEP teflon thermal blanket specimens.

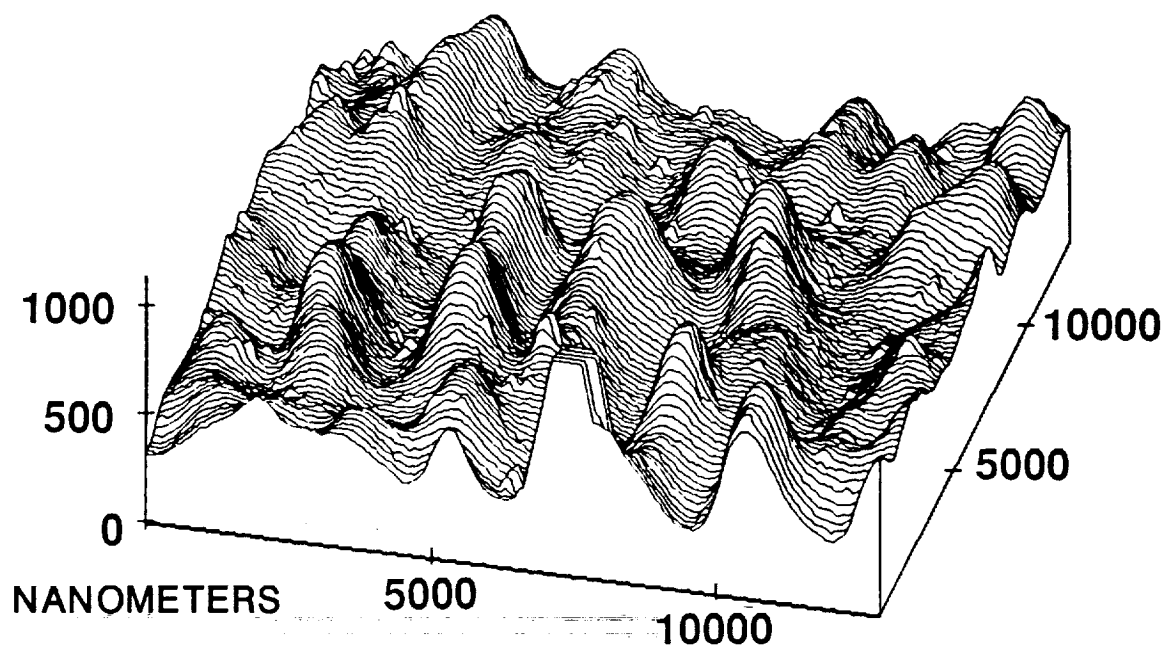
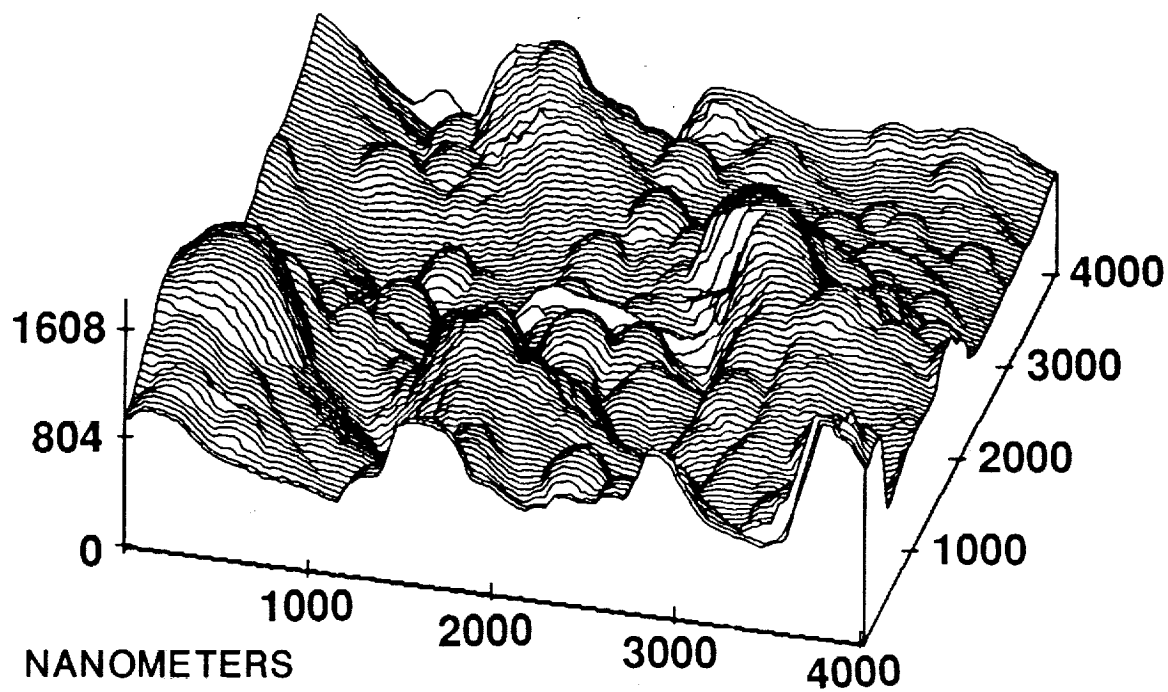
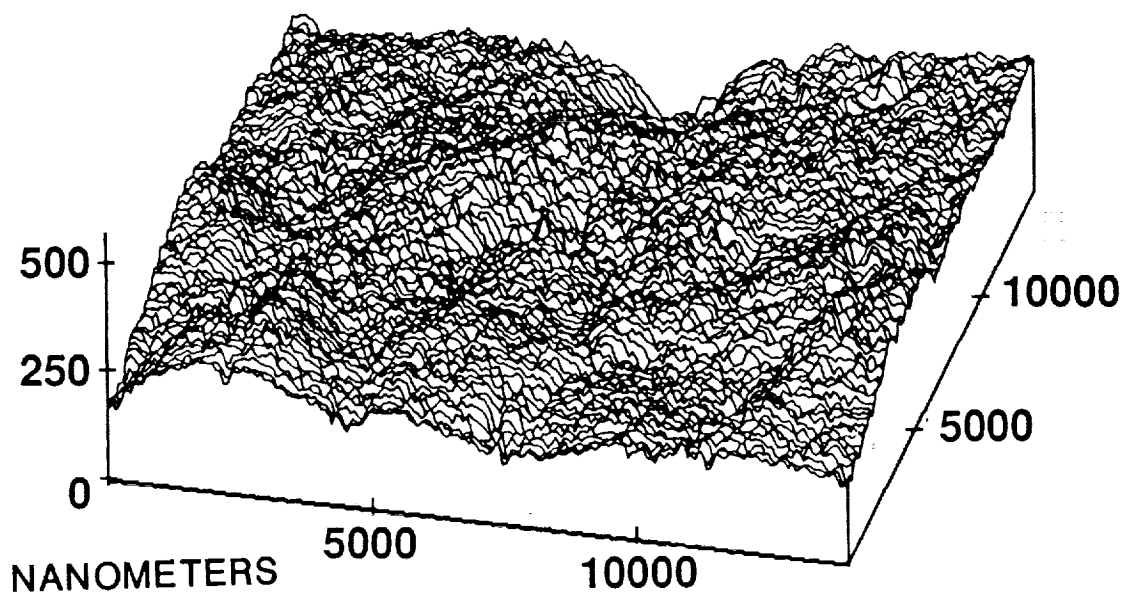


Figure 12. STM line plot analysis of LDEF exposed F2 silvered FEP teflon thermal blanket specimen.



(a) LDEF exposed C8 (opaque).



(b) After rubbing (specular).

Figure 13. STM line plot analysis of (a) C8 silvered FEP teflon thermal blanket specimen and (b) specimen shown in (a) after rubbing surface with a spatula.

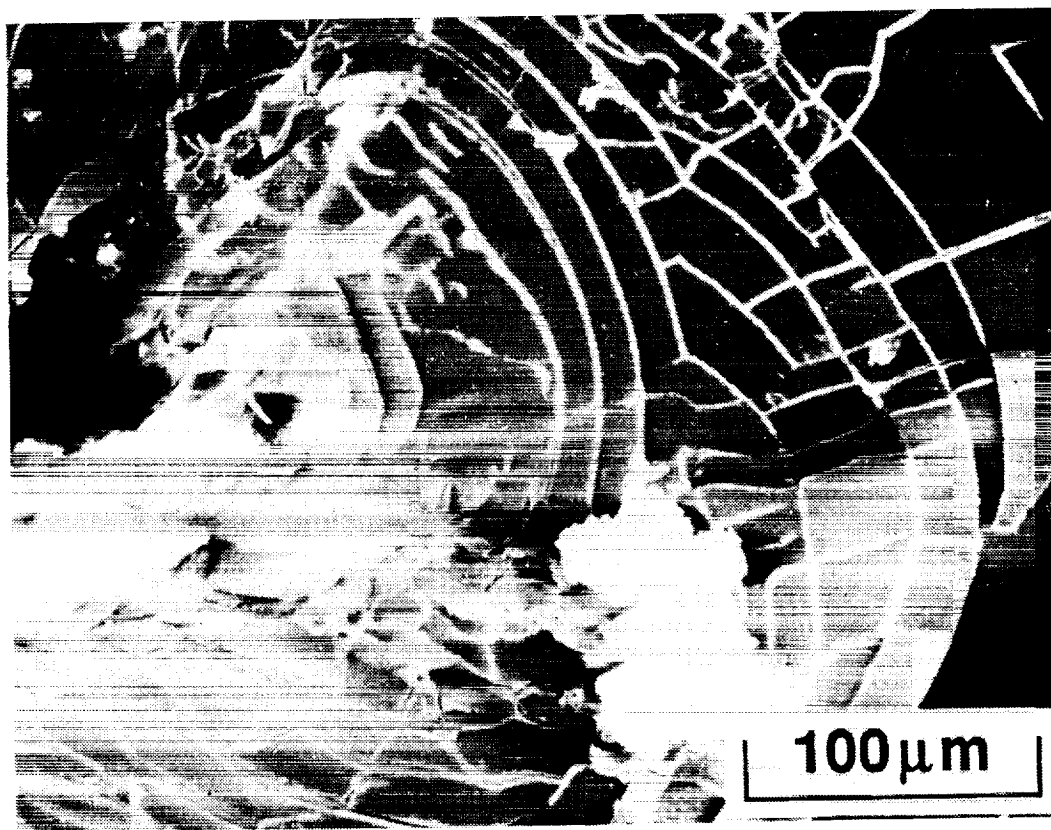


Figure 14. SEM of contaminated portion of the A10 thermal blanket.

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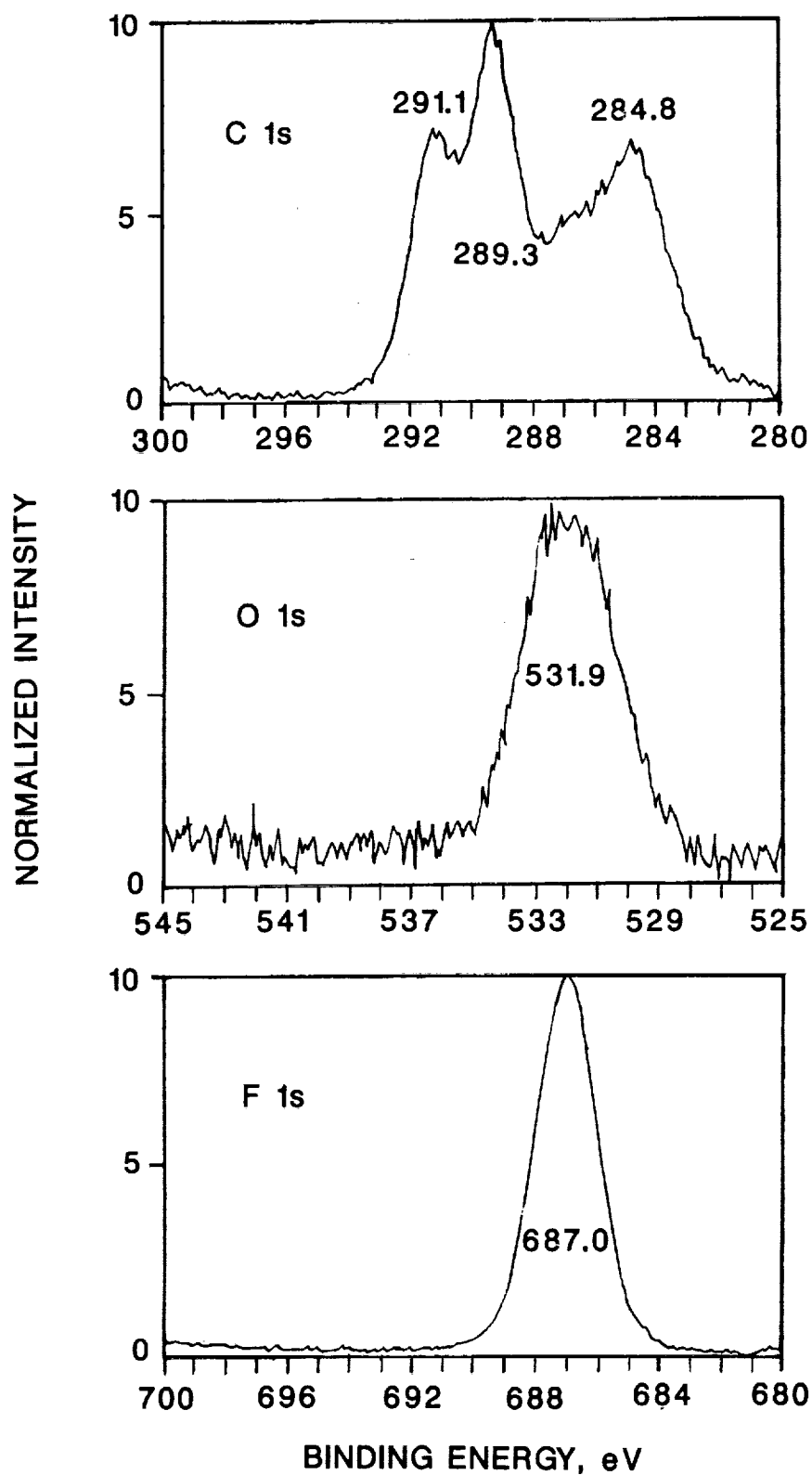


Figure 15. Narrow scan XPS spectra for C5 thermal blanket specimen.

REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
Public reporting burden for this collection of information is estimated to average 1 hour per response, including the time for reviewing instructions, searching existing data sources, gathering and maintaining the data needed, and completing and reviewing the collection of information. Send comments regarding this burden estimate or any other aspect of this collection of information, including suggestions for reducing this burden, to Washington Headquarters Services, Directorate for Information Operations and Reports, 1215 Jefferson Davis Highway, Suite 1204, Arlington, VA 22202-4302, and to the Office of Management and Budget, Paperwork Reduction Project (0704-0188), Washington, DC 20503.				
1. AGENCY USE ONLY(Leave blank)	2. REPORT DATE December 1991	3. REPORT TYPE AND DATES COVERED Technical Memorandum		
4. TITLE AND SUBTITLE An Analysis of LDEF-Exposed Silvered FEP Teflon Thermal Blanket Material		5. FUNDING NUMBERS WU 506-43-11		
6. AUTHOR(S) Philip R. Young Wayne S. Slemph				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) NASA Langley Research Center Hampton, VA 23665-5225		8. PERFORMING ORGANIZATION REPORT NUMBER		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) National Aeronautics and Space Administration Washington, DC 20546-0001		10. SPONSORING/MONITORING AGENCY REPORT NUMBER NASA TM-104096		
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Unclassified-Unlimited Subject Category 27		12b. DISTRIBUTION CODE		
13. ABSTRACT (Maximum 200 words) The characterization of selected silvered fluorinated ethylene propylene (FEP) teflon thermal blanket material which received 5 years and 9 months of exposure to the LEO environment on the Long Duration Exposure Facility is reported. X-ray photoelectron spectroscopy, infrared, and thermal analyses did not detect a significant change at the molecular level as the result of this exposure. However, various microscopic analyses revealed a roughening of the coating surface due to atomic oxygen erosion which resulted in some materials changing from specular reflectors of visible radiation to diffuse reflectors. The potential effect of silicon-containing molecular contamination on these materials is addressed.				
14. SUBJECT TERMS LDEF, Environmental Exposure, Silvered FEP Teflon, Atomic Oxygen, Chemical Characterization		15. NUMBER OF PAGES 27		
		16. PRICE CODE A03		
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT	

NSN 7540-01-280-5500

Standard Form 298 (Rev. 2-89)
Prescribed by ANSI Std. Z39-18
298-102

NASA-Langley, 1991